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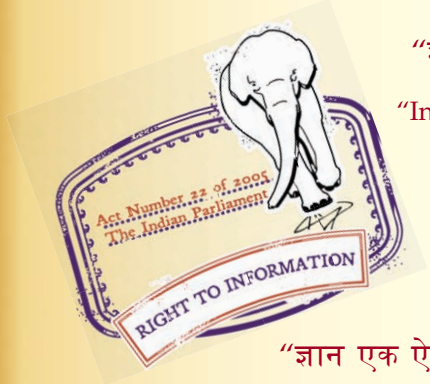

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IS : 7220 - 1974
(Reaffirmed 1995)

Indian Standard

SPECIFICATION FOR ETHYLENEDIAMINETETRA-ACETIC ACID, PURE AND TECHNICAL

(First Reprint OCTOBER 1999)

UDC 547.415.1'292

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

December 1974

AMENDMENT NO. 1 DECEMBER 1995
TO
IS 7220 : 1974 SPECIFICATION FOR ETHYLENEDIAMINETETRA-ACETIC ACID, PURE AND TECHNICAL

[*Page* 4, *Table* 1, *Sl No.* (i), *col* 3 *and* 4] — Substitute '98.0' *for* '98 (Pure grade)' and '96.0' *for* '96 (Technical grade)'.

[*Page* 4, *Table* 1, *Sl No.* (ii), *col* 3 *and* 4] — Substitute '0.10' *for* '0.1 (Pure grade)' and '0.50' *for* '0.5 (Technical grade)'.

[*Page* 4, *Table* 1, *Sl No.* (vi), *col* 3] — Substitute '0.30' *for* '0.3'.

(*Page* 5, *clause* A-2.1.3) — Substitute '0.1 M' *for* '0.1 N.'

(*Page* 6, *clause* A-2.2.1, *lines* 1 *and* 2) — Substitute 'zinc sulphate 7 H_2O.' *for* 'zinc sulphate' and '5 ml of buffer solution' *for* '50 ml of buffer solution'.

(PCD 9)

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[*Page* 6, *clause* A-2.2.1, *line* 2 (*see also Amendment No.* 1)] — Substitute '5 to 10 ml' *for* '50 ml'.

(PCD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR ETHYLENEDIAMINETETRA-ACETIC ACID, PURE AND TECHNICAL

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Indian Standard

SPECIFICATION FOR ETHYLENEDIAMINETETRA-ACETIC ACID, PURE AND TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 11 January 1974, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 In the preparation of this standard substantial assistance has been derived from TGL 8411 'Specification for ethylenediaminetetra-acetic acid'; Reagents Chemicals and Standards, 3rd Edition; and the data supplied by Hico Products Private Limited, Bombay.

0.3 Ethylenediaminetetra-acetic acid, is produced by hydrolysis of nitrile of ethylenediaminetetra-acetic acid. It is used for pharmaceutical purposes and in various processing industries like textiles, chemicals, cosmetics, detergents, paper, paints and electroplating.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for ethylenediaminetetra-acetic acid, pure and technical.

2. GRADES

2.1 The material shall be of two grades, namely, pure grade and technical grade.

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — The material shall be white or slightly bluish, crystalline powder. The material is almost insoluble in cold water, alcohol and in the usual organic solvents. It is easily soluble in aqueous solutions of alkalis, ammonia and alkali carbonates.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in column 5 of Table 1.

TABLE 1 REQUIREMENTS FOR ETHYLENEDIAMINETETRA-ACETIC ACID

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Pure Grade	Technical Grade	
(1)	(2)	(3)	(4)	(5)
i)	Purity, percent by mass, *Min*	98	96	A-2
ii)	Sulphated ash, percent by mass, *Max*	0·1	0·5	A-3
iii)	Iron (as Fe), percent by mass, *Max*	0·002	0·005	A-4
iv)	Heavy metals (as Pb), percent by mass, *Max*	0·001	0·001	A-5
v)	Solubility	To pass the test		A-6
vi)	Nitrilotriacetic acid content, percent by mass, *Max*	0·3	—	A-7

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in brown, wide neckbottles with threaded cover, tightly screwed and/or in polythene lined gunny bags and shall be kept in a cool and dry place protected from light.

4.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and grade of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Lot or batch number in code or otherwise; and

d) Gross, net and tare mass.

4.2.1 The product may also be marked with Standard mark.

4.2.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to this standard determined by the methods prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR ETHYLENEDIAMINETETRA-ACETIC ACID

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. ASSAY

A-2.1 Reagents

A-2.1.1 *Strong Ammonium Hydroxide Solution* — approximately 27 to 30 percent (*v/v*).

A-2.1.2 *Buffer Solution* — Dissolve 67·5 g of ammonium chloride in 650 ml of strong ammonia solution and add sufficient water to produce 1 000 ml.

A-2.1.3 *Standard Zinc Sulphate Solution* — 0·1 N. Prepare by weighing 28·756 g of zinc sulphate, dissolving it in water and making up the volume to 1 000 ml in a 1 000-ml graduated flask (*see* IS : 915-1958†).

*Specification for water, distilled quality (*revised*).
†Specification for one-mark graduated flasks.

A-2.1.4 *Eriochrome Black T Indicator* — Dissolve 0·1 g of eriochrome black T in 100 ml of water containing a few drops of the buffer solution.

A-2.2 Procedure — Weigh accurately about 2 g of the material and dissolve it in water by adding a few drops of alkali (*p*H 8·5). Dilute with water to make up the volume to 100 ml in a 100-ml graduated flask (*see* IS : 915-1958*). Fill the burette with this solution (solution A).

A-2.2.1 Pipette out 10 ml of the standard zinc sulphate solution in a 250-ml conical flask. Add to it 50 ml of buffer solution and 2 drops of eriochrome black T indicator. Titrate it against solution A till the final blue colour is obtained.

A-2.3 Calculation

$$\text{Purity, percent by mass} = \frac{2\,922{\cdot}5}{V \times M}$$

where

V = volume in ml of the solution A (*see* **A-2.2**), and
M = mass in g of the material taken for the test (*see* **A-2.2**).

A-3. DETERMINATION OF SULPHATED ASH

A-3.1 Apparatus

A-3.1.1 *Platinum Crucible*

A-3.1.2 *Muffle Furnace* — capable of maintaining temperature of 900 ± 25°C.

A-3.2 Reagents

A-3.2.1 *Sulphuric Acid* — conforming to IS : 266-1961†.

A-3.3 Procedure — Weigh accurately about 20 g of the material in a platinum crucible, ignite and heat the material in the muffle furnace at 900°C. When most of the material has been burnt, remove the platinum crucible. Allow it to cool and then add 3 to 4 drops of sulphuric acid. Heat again in the furnace for one hour. Cool to room temperature and weigh.

A-3.4 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100\,M_1}{M_2}$$

where

M_1 = mass in g of the ignited residue, and
M_2 = mass in g of the material taken for the test.

*Specification for one-mark graduated flasks.
†Specification for sulphuric acid (*revised*).

A-4. TEST FOR IRON

A-4.1 Apparatus

A-4.1.1 *One-Mark Graduated Flasks* — 50 ml, 100 ml, 1 000 ml (*see* IS : 915-1958*).

A-4.1.2 *Nessler Cylinders* — 50 ml (*see* IS : 4161-1967†).

A-4.2 Reagents

A-4.2.1 *Hydrochloric Acid* — conforming to IS : 265-1962‡.

A-4.2.2 *Nitric Acid* — conforming to IS : 264-1968§.

A-4.2.3 *Dilute Hydrochloric Acid* — approximately 1 N.

A-4.2.4 *Potassium Permanganate Solution* — approximately 0·02 N.

A-4.2.5 *Ammonium Thiocyanate Solution* — Dissolve 20 g of ammonium thiocyanate in 100 ml of water.

A-4.2.6 *Standard Iron Solution* — Weigh 0·702 2 g of ferrous ammonium sulphate [$(NH_4)_2SO_4.FeSO_4.6H_2O$] into a 1 000-ml graduated flask and dissolve in 100 ml of water. Add 5 ml of dilute sulphuric acid (1 : 5 *v/v*) and run in continuously potassium permanganate solution until a slight pink colouration remains after stirring well. Make up the volume to 1 000 ml with water and mix thoroughly. Pipette out 10 ml of this solution into a 100-ml graduated flask and make up the volume to 100 ml with water. Mix thoroughly. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-4.3 Procedure — Weigh accurately 5 g of the material and ignite it according to procedure prescribed under **A-3.3**. Add 4 ml of hydrochloric acid, 4 ml of water and 1 ml of nitric acid and slowly evaporate to dryness on steam-bath. Add to the residue 1 ml of dilute hydrochloric acid, 40 ml of water and digest for 5 minutes. Cool and transfer the solution quantitatively to the 50-ml graduated flask and make up the volume with water to the mark. *Reserve this solution for tests under* **A-4.3.1** *and* **A-5.3**.

A-4.3.1 Transfer 10 ml of the solution (**A-4.3**) to the Nessler cylinder and add 20 ml of water. Add 2 ml of hydrochloric acid, potassium permanganate solution drop by drop till a faint pink colour remains, and 5 ml of ammonium thiocyanate solution. Mix thoroughly and make up the volume with water to 50 ml. Take in another Nessler

*Specification for one-mark graduated flasks.
†Specification for Nessler cylinders.
‡Specification for hydrochloric acid (*revised*).
§Specification for nitric acid (*revised*).

cylinder 20 ml of water, 2 ml of hydrochloric acid, and following quantities of standard iron solutions:

a) 2 ml of standard iron solution in the case of the pure grade, and
b) 5 ml of standard iron solution in the case of the technical grade.

Add two drops of permanganate solution and then 5 ml of ammonium thiocyanate solution, mix thoroughly, make up the volume to 50 ml with water and then compare the colours of both solutions.

A-4.3.2 The limits prescribed for iron in Table 1 shall be regarded as not having been exceeded if the intensity of red colour produced with the material is not deeper than that produced with standard iron solutions.

A-5. TEST FOR HEAVY METALS

A-5.1 Apparatus

A-5.1.1 *One-Mark Graduated Flask* — 1 000 ml (*see* IS : 915-1958*).

A-5.1.2 *Nessler Cylinders* — 50 ml (*see* IS : 4161-1967†).

A-5.2 Reagents

A-5.2.1 *Hydrogen Sulphide Solution* — freshly prepared saturated aqueous solution.

A-5.2.2 *Standard Lead Solution* — Weigh 1·60 g of lead nitrate [$Pb(NO_3)_2$] into a 1 000-ml graduated flask and dissolve in water. Make up the volume to 1 000 ml with water. Further pipette out 10 ml of this solution into another 1 000-ml graduated flask and dilute it with water to make up the volume to 1 000-ml. One millilitre of the resulting solution contains 0·01 mg of lead (as Pb).

A-5.2.3 *Dilute Hydrochloric Acid* — approximately 1 N.

A-5.3 Procedure — Pipette out 20 ml of solution reserved under **A-4.3** into the Nessler cylinder. Dilute it with water to 35 ml and then add 10 ml of hydrogen sulphide solution. Make up the volume to 50 ml with water. Carry out a control test in another Nessler cylinder with 2 ml of standard lead solution, adding 0·3 ml of dilute hydrochloric acid and 10 ml of hydrogen sulphide solution and make up the volume to 50 ml with water. Mix thoroughly both the solutions and compare the colours produced.

A-5.3.1 The limits prescribed for heavy metals in Table 1 shall be regarded as not having been exceeded if the intensity of colour produced in the test with the material is not deeper than that produced in the control test.

*Specification for one-mark graduated flasks.
†Specification for Nessler cylinders.

A-6. DETERMINATION OF SOLUBILITY

A-6.1 Reagent

A-6.1.1 *Sodium Hydroxide Solution* — 20 percent (*m/v*).

A-6.2 Procedure — Weigh accurately 30 g of the material into a 250-ml beaker. Treat it with 100 ml of the sodium hydroxide solution and examine the resulting solution.

A-6.2.1 The material shall be regarded as having passed the test if the resulting solution is bright and colourless.

A-7. DETERMINATION OF NITRILOTRIACETIC ACID CONTENT

A-7.1 Apparatus

A-7.1.1 *An Automatic Recording Polarograph*

A-7.2 Reagents

A-7.2.1 *Potassium Hydroxide Solution* — 10 percent (*m/v*).

A-7.2.2 *Ammonium Nitrate Solution* — 10 percent (*m/v*).

A-7.2.3 *Cadmium Nitrate Solution* — 3 percent (*m/v*).

A-7.2.4 *Eriochrome Black T Indicator Solution* — Dissolve 0·1 g of the dye in 20 ml of rectified spirit conforming to IS : 323-1959*. This solution shall be prepared fresh every week.

A-7.2.5 *Methyl Red Indicator Solution* — Dissolve 25 mg of methyl red in 100 ml ethyl alcohol (60 percent *v/v*).

A-7.2.6 *Standard Stock Solution* — Dissolve 1·0 g of nitrilotriacetic acid in 10 ml of potassium hydroxide solution in a 100 ml volumetric flask, dilute to mark with water, and mix.

A-7.2.7 *Sample Solution* — Dissolve 10·0 g of the sample in 87 ml of potassium hydroxide solution in a 100-ml volumetric flask, dilute to the mark with water and mix.

A-7.2.8 *Standard Solution* — Mix 10·0 ml of the sample solution with 1·0 ml of the standard stock solution in a 100-ml volumetric flask, dilute to the mark with water and mix.

A-7.2.9 *Test Solution* — Transfer 10·0 ml of the sample solution to a 100-ml volumetric flask, dilute to volume with water and mix.

A-7.3 Procedure — To 20 ml of the standard solution (**A-7.2.8**) in a 150-ml beaker, add 1 ml potassium hydroxide solution, 2 ml of ammonium

*Specification for rectified spirit (*revised*).

nitrate solution and 10 ml of eriochrome black T indicator sufficient to impart colour to the solution. Titrate with cadmium nitrate solution to a red end-point. Record the titre value and discard the solution. To another 20 ml aliquot of the solution in a 100-ml volumetric flask, add a volume of cadmium nitrate solution equal to the volume titrated, and add 0·05 ml in excess. Add 1·5 ml of potassium hydroxide solution, 10 ml of ammonium nitrate solution and 0·5 ml of methyl red indicator solution, dilute to volume with water and mix. Transfer a portion of this solution to a polarographic cell and de-aerate by bubbling nitrogen through the solution for 10 minutes. Insert the dropping mercury electrode of the polarograph, and record the polarogram from −0·6 volt to −1·2 volt at a sensitivity of 0·006 μA/mm, using a saturated calomel electrode. Similarly repeat the test with the test solution.

A-7.3.1 The material shall be taken to have passed the test if the diffusion current for the test solution is not greater than 30 percent of the difference between the diffusion currents for the standard solution and the test solution (0·3 percent nitrilotriacetic acid).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF ETHYLENEDIAMINETETRA-ACETIC ACID

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The sample shall be placed in clean, dry and airtight glass containers or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, batch number and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type undivided sampling tube, (*see* Fig. 1) consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel), one closely fitting into the other throughout their length so that it is possible to rotate one tube within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference area cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with a locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm, proportionately to the length. A length of 150 cm and a diameter of 30 cm can cater for most needs.

B-2.1.1 *Use of Sampling Instrument* — The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same container such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

B-3. SCALE OF SAMPLING

B-3.1 Lot — All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to

the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected for drawing the samples shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

Lot Size (N)	No. of Containers to be Selected (n)
(1)	(2)
4 to 25	3
26 „ 50	4
51 „ 100	5
101 „ 150	6
151 „ 300	7
301 and above	8

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

B-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used (*see* IS : 4905-1968*). In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3, ..., up to r, and so on, where r is the integral part of N/n (N being the lot size and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to give sample for test.

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From each of the containers selected as in **B-3.2**, draw with the sampling instrument of an appropriate size small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

*Methods for random sampling.

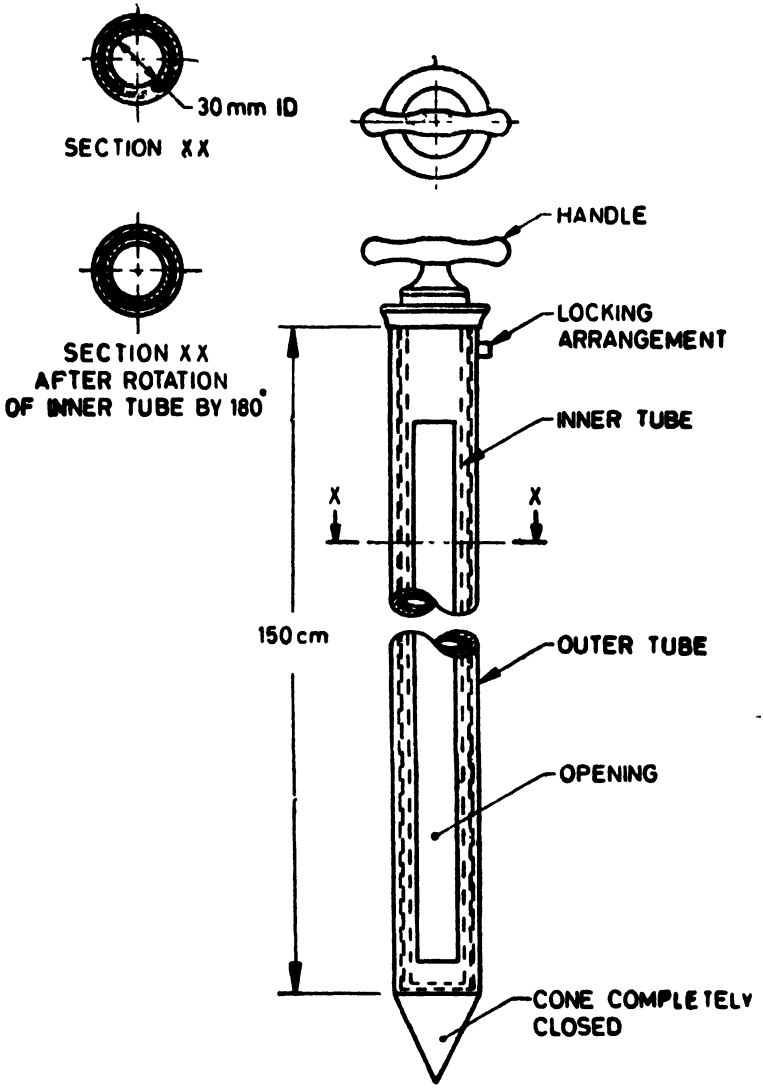

FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

B-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

B-4.3 From the samples (**B-4.2**) representing different containers selected in **B-3.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient to carry out testing for the characteristics specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-4.4 The remaining portion of the material in the samples (**B-4.2**) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-4.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars given in **B-1.7**.

B-4.6 The referee samples consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-5. TESTS

B-5.1 Tests for description and purity shall be conducted on each of the individual samples.

B-5.2 Tests for the determination of all other characteristics shall be conducted on the composite sample.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of description and purity if each of the test results satisfy the corresponding requirement given in **3.1** and Table 1.

B-6.2 For Composite Samples — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample (*see* **B-5.2**), the test results shall satisfy the relevant requirements given in Table 1.

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Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G. T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-58C, L. N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
Patliputra Industrial Estate, PATNA 800013	26 23 05
T. C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17
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